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DETROIT

The Cold-Cathode Gas Diode

Special circuits and improved voltage characteristics may make this component useful in computers

WITH the increasing use of electronic digital computers, complex problems have been presented whose solutions require high-speed memories so large that present-day costs make them economically impractical. The cold-cathode gas diode—such as the simple neon glow lamps—offers possibilities for low-cost high-speed memories, once the inherent disadvantages of the tubes are overcome. A. W. Holt, D. C. Friedman, and W. D. Urban of the National Bureau of Standards data processing systems laboratory have undertaken two separate studies of these visual-indicating tubes. The result has been a method of improving gas diode voltage characteristics and a number of circuits that provide an approach to reducing the cost of digital computer memories from the present dollar per bit (binary digit) to about 10 cents. In addition to the storage properties of these gas-diode memory circuits, indication is also possible since in most of the circuits considered the lamp will flash in only one of the two stored states. The NBS studies also disclosed that gas diode voltage characteristics can be improved by an aging process. Both investigations were sponsored by the Wright Air Development Center and the Air Force Cambridge Research Center.

The cold-cathode gas diode has many qualities which would make it an excellent computer component. It requires little power, but it can handle large currents momentarily. It is small, light, rugged, cool in operation, and inexpensive. Life for normal indicator operation is over 10,000 hours. Its operation may be stated binary-wise in several ways, so that it provides the YES–NO statement of a digital computer element. In electronic computer circuits, with few passive elements, it may be used as an and-gate, an or-gate, an indicator-type memory bit, a flip-flop, an oscillator, or an indicator. It lends itself to visual or photoelectrical output, as well as electrical output of either polarity.

However, four major defects in the operation of this component have thus far limited its use. Because it is manufactured as an indicator, only one characteristic—the maximum firing voltage—is controlled, and this may have a large tolerance. This leads to the first

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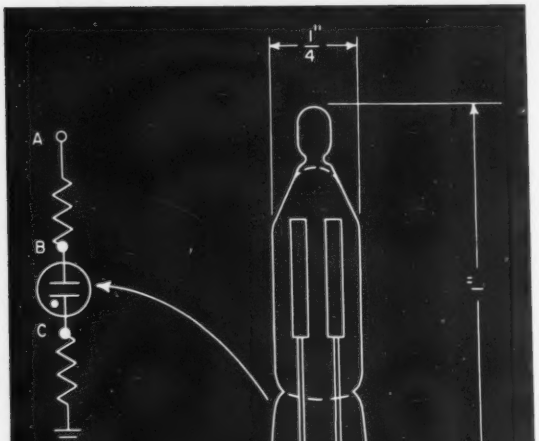


Basic gas diode circuit. This circuit may be a pilot lamp, an oscillator, a flip-flop, an or-gate, an and-gate, a memory bit, or even a photo-flash. How it is used depends upon the voltages applied to points A, B, and C, and the values of the resistors.

defect: In a batch of tubes, a wide range in firing and holding voltages is commonly found. Selection might be used to overcome this difficulty, except for the second defect: The characteristics of any one tube vary unpredictably with use. The other two major defects are associated with use and circuitry: The normally long deionization time limits speed of operation, and the need for a continuous path to maintain ionization makes selection circuits difficult to engineer.

Gas Diode Circuits

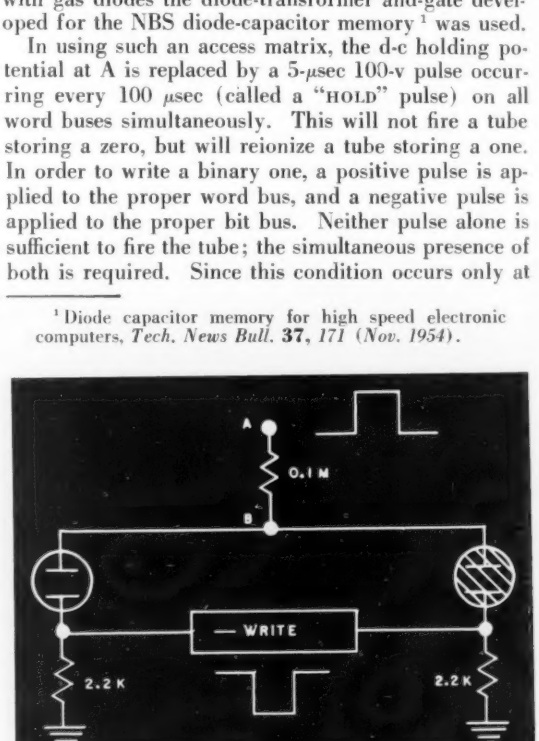
Basic Circuit. The basic memory circuit consists simply of a gas diode connected in series between two resistors. Power is applied to one resistor at a point which may be called A for convenience. This resistor is connected to the diode at point B. The other terminal of the diode is tied, at point C, to the second resistor which is returned to ground. This circuit may be a pilot lamp, an oscillator, a flip-flop, an or-gate, an and-gate, a memory bit, or even a photo-flash. How it is used depends upon the voltages applied to points A, B, and C, the values of the resistors, and whether the output is electrical, photoelectrical, or optical.

When used as a d-c indicator memory bit, the total resistance exclusive of the diode is of the order of 0.1 megohm. A potential somewhere between the firing voltage and the holding voltage is applied at point A. If the tube is unfired, this potential is not enough to cause conduction, and the bulb will be dark. This may be taken as the binary zero. B is at the supply voltage; C is at ground. If now a positive potential is applied momentarily at A or B, or a negative potential is applied at C, the tube may be caused to conduct. This makes it glow. Points B drops in potential while point C rises. The new levels will be maintained as long as the potential at A is above the minimum holding potential for the tube. This may be considered as the binary one. To return to the binary zero state, it is necessary to interrupt the holding potential until the tube deionizes fully. This is one disadvantage of the circuit; another is that a continuous path is necessary to maintain the tube in the binary-one state, making it difficult to obtain access to one bit without disturbing others.

Memory Access Circuits. The first innovation was an attempt to overcome the access difficulties. It consisted of turning from the static bit just described to a dynamic type of storage. In this system, a binary one is stored if a tube has "recently" been fired. "Recently," in this sense, means within the last 100 microseconds. A binary zero is stored if the tube has not been fired in this time. Since a continuous path need not be maintained for storage, any scheme of matrix access circuitry may be employed. In the experiments with gas diodes the diode-transformer and-gate developed for the NBS diode-capacitor memory [1] was used.

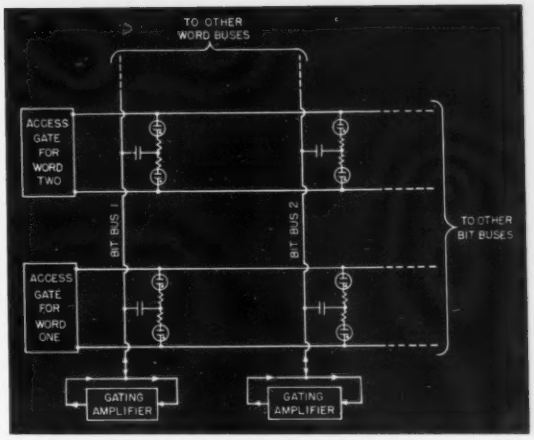
In using such an access matrix, the d-c holding potential at A is replaced by a 5-μsec 100-v pulse occurring every 100 μsec (called a "HOLD" pulse) on all word buses simultaneously. This will not fire a tube storing a zero, but will reionize a tube storing a one. In order to write a binary one, a positive pulse is applied to the proper word bus, and a negative pulse is applied to the proper bit bus. Neither pulse alone is sufficient to fire the tube; the simultaneous presence of both is required. Since this condition occurs only at

[1] Diode capacitor memory for high speed electronic computers, *Tech. News Bull.* **37**, 171 (*Nov. 1954*).



The double-diode memory circuit. This type of memory may be used generally as a high-speed memory. It is an indicating type of memory which may be read out visually by hiding one bulb of each pair or read out photoelectrically, with either push-pull or on-off input to the photocells.

Organization of the double gas-diode-capacitor-memory. This circuit is a variation of the NBS junction-type-diode-capacitor memory. Here the semiconductor diodes are replaced by gas diodes. The gas diodes either clamp one side of the storage capacitor or disconnect it.



the intersection of the selected buses, only the desired bits are written as ones.

In order to examine the stored words, a positive READ pulse is applied to the proper word bus. If the tube has been ionized recently, this pulse is sufficient to cause conduction, and a signal will be sensed at the READ REPEATER. During the holding pulse, the logical sum of the memory appears at the READ REPEATERS if this information is desired.

This is a one-way memory: That is, information that has been read into the memory must be completely erased before new information can be read in. In order to write a zero, it is necessary to inhibit the HOLD pulse until the tube will no longer reionize when it is applied, and no provision has been made to make this occur on a single bit. However, because it is a high-speed one-way memory, it has several possible applications. For example, it may be used as a buffer between the internal memory of a computer and a high-speed printer. In it would be stored all the information for a long print-out. While the printing operation is going on, the computer may be calculating the next set of data. An erasing time of about one millisecond may be economically allowed at the end of a printing cycle in this application. The memory may also be used as a visual tally board for a high-speed computer. In this case, erasure will be controlled by the operator at suitable intervals.

Double-Diode Memory Circuits. The second innovation was made to provide high writing speed in both directions, using a double-diode bit. In this circuit, two gas diodes, three resistors, and a negative WRITE source are employed. Both tubes are fed from the power source through a common resistor. The power source is at the A end of the common resistor, and the anodes of both diodes are tied to the B end of the resistor. The cathodes of the diodes are returned to ground through separate resistors. The cathodes are also connected to the outputs of the negative WRITE source.

In the operation of this circuit, a d-c potential above the firing potential is applied to A. One of the bulbs will thus always be on. To write in the opposite digit, the cathode of the "off" tube is pulled down with a negative WRITE pulse, which causes it to fire. This causes the common point B to drop to near ground potential, so that the "on" bulb now has less than holding voltage across it. It goes out and starts to deionize. At the end of the WRITE pulse the point B rises again to holding potential, but all the current goes through the newly "on" bulb while the other continues toward its fully deionized state. Since the system is symmetrical, fast writing is possible in either direction.

Laboratory studies show that changing from a one to a zero, or vice versa, may be accomplished in 5 microseconds or less. However, it takes some time to reach steady-state conditions; so that as the repetition rate of changing from a one to a zero and back again increases, it becomes more difficult to determine whether switching has taken place. At the limiting rate, the "off" and "on" tubes have the same state of conduction.

This type of memory may be used generally as a high-speed memory, provided the rapidity of change of any one datum is limited to the speed of changing from one binary state to the other. It is an indicating type of memory which may be read out visually by hiding one bulb of each pair or read out photoelectrically, with either push-pull or on-off input to the photocells.

Gas Diode-Capacitor Memory. Another memory which operates more slowly in one direction than the other has been the subject of limited study. Here, a gas diode is connected in series with a capacitor. Bipolar pulses are applied to the other side of the diode, and READ-WRITE pulses are applied to the other end of the capacitor. Suppose that the capacitor has been charged to -15 v. When the first half of a bipolar pulse is applied to the top terminal of the diode, there is sufficient voltage across the diode to fire it. Conduction takes place and the capacitor tries to charge toward $+15$ v. Whether it reaches this potential is immaterial, since during the second portion of the bipolar pulse the tube conducts in the opposite direction and charging is toward -15 v. At the end of charging, the capacitor is left with the original charge. Thus a binary one (since the tube flashed) has been read and restored. If, however, the capacitor had been uncharged, the bipolar pulse would not have fired the bulb, the tube would have remained dark, and the capacitor charge would have remained unchanged. Since in this case all leakage would be toward ground, there is no chance of losing a zero. However, a binary one would tend to leak toward zero and therefore must be regenerated by the process of reading.

In order to write a binary one, it is necessary only to fire the tube during the bipolar pulse. This may be done by a negative bottom WRITE pulse applied during the first half of the bipolar operating pulse. The regu-

lar regeneration cycle then holds this new information. To write a binary zero, the tube is fired during the first portion of the bipolar pulse; then, when the pulse starts down to charge the capacitor in the opposite direction, a bottom pulse is applied which holds the lower plate of the capacitor at −15 v. This does not prevent the normal cycle of charging the upper plate to −15 v, since this potential is set by the operating pulse amplitude and the drop across the diode. The bottom pulse is maintained until the upper pulse is over. At this time the upper plate of the capacitor is disconnected so that when the bottom is returned to ground, the top also rises to ground potential. Deionization must have progressed sufficiently before the next operating pulse to prevent refiring of the bulb.

Other Memory Circuits. A variation of the NBS diode-capacitor memory (see footnote 1) can be achieved by replacing the semiconductor diodes with gas diodes. Here the diodes are used only to clamp one side of the storage capacitor or to disconnect it. This makes the requirements concerning the characteristics of the diodes much less stringent. In the operation of this circuit, the capacitor is charged a few volts positive for a binary one and a few volts negative for a binary zero. To write a one, the diodes are fired, clamping the top of the capacitor to approximately ground, and the bottom is pulled downward to −25 v. When the clamping pulses are terminated, the diodes deionize disconnecting the top of the capacitor. The bottom pulse is then released, raising the top of the capacitor to +25 v. Similarly, to write a zero, a positive bottom write pulse is used.

Reading is accomplished by clamping the top of the capacitor and noting the polarity of the pulse at the input to the read amplifier. To restore the information, the bottom pulse is applied in the same polarity as the pulse obtained upon clamping. Regeneration is necessary because of the destructive reading operation as well as leakage. Since the bit amplifiers are used in common, regeneration must be done word by word.

Although this is not primarily an indicator type of memory, it is possible to make it one. When operated in the mode just described, one diode carries more current than the other during rewriting, depending upon the polarity of rewriting, and this may be noted photoelectrically. In another mode of operation, regeneration is accomplished by releasing one clamping diode immediately after sensing. This probably would be noticeable even visually. Writing may be accomplished in 10 to 50 μsec.

High firing voltages across the diode may be dispensed with by supplying only low voltage to the diodes for charging purposes and a radiofrequency voltage to a shield about the tube to fire it.

Improving Gas Diode Voltage Characteristics

An inexpensive method for equalizing and stabilizing the voltage characteristics of cold-cathode gas diodes has been developed by the data processing systems laboratory. The tube characteristics are improved by a process of aging in which pulsed voltages are applied simultaneously to a large number of tubes. Trials of the processed tubes in actual operating circuits show that these aged values are reasonably stable.

It is, of course, possible to obtain special tubes which are stable and generally equal in characteristics. However, these tend to be costly. The aim of this investigation has been to devise a method to obtain high-quality gas diodes by inexpensive processing of low-cost indicator lamps. Therefore, the bulk of the work was done with NE–2 and NE–51 lamps taken from general stock.

The circuit for the equalizing process consists of a large number of tubes in parallel connected to a pulse power source through a common resistor. Pulses are continually applied until the firing potentials and holding potentials of all the tubes rise to common terminal values. Further processing produces no additional change. In the stability check, all the tubes are subjected to identical conditions. Providing a separate

Gas diode tube aging racks used at NBS for equalizing and stabilizing voltage characteristics for type NE–2 and NE–51 tubes. The circuit for the equalizing process consists of a large number of tubes in parallel connected to a pulse power source through a common resistor.

Voltage characteristic tester used to check firing and holding potentials of gas diodes before, during, and after aging. Equalizing and stabilizing tube voltage characteristics promise to provide an inexpensive, easily available component for many practical applications in automatic electronic computor applications.

resistor for each tube minimizes mutual interaction, while the power supply and environmental changes are common to all tubes.

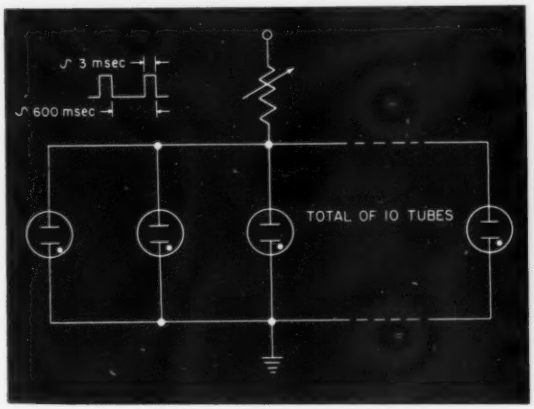
The first characteristic potential to be considered is the firing voltage—the minimum voltage necessary to start conduction in a tube which has been off a long time compared to its deionization time. The second is the holding or maintaining voltage—the voltage drop across the tube when it is carrying its rated current. For the NE–2, these are about 90 and 75 volts, respectively. For the NE–51, these values are approximately 5 volts lower. Both tubes are rated at 1/25 watt.

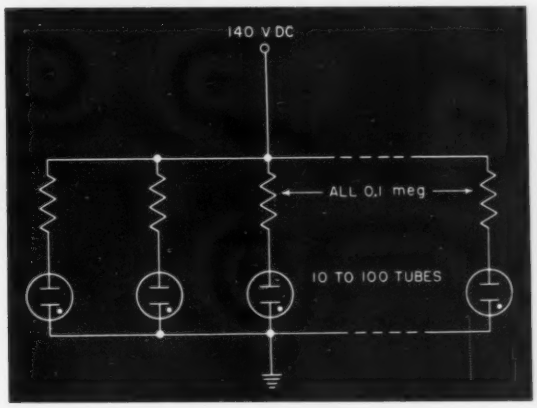
Since equalizing the tubes consists mainly of operating the tubes beyond their normal operating range, some means of obtaining pulsed operation was required. Two methods were used here. For very large overloads, a relay pulser was employed. The length of the pulse was determined by the break time of a set of relay contacts. When desired, this time could be lengthened by use of an RC circuit, but usually it was held to a minimum. The "off" time was set by a timing relay also operated with a variable RC circuit. Two relays were used to obtain "fail-safe" operation insofar as the tubes were concerned. For tests at higher duty cycles, full or half-wave 60-cycle voltage was used. To keep the over-all duty cycle low, a clock-driven switch was used to obtain 25- or 75-percent duty cycles over approximately 100-second periods. In all tests, bulb temperature was checked to make sure it did not rise significantly above ambient temperature.

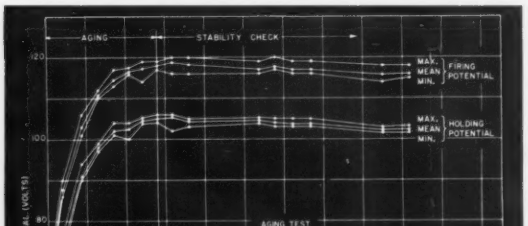
After some preliminary tests, a setup was devised for equalizing the characteristics of 10 tubes at a time. This was based on the assumption that at some time during the life of the tubes, when used as indicators, the characteristic voltages must rise, since end of life is indicated by the voltages rising to the point where the tubes will not operate in the circuit. It was also assumed that stability could not be attained until the tube characteristics had started on this rise. The 10 tubes were connected in parallel and fed from the pulse source through a common resistor.

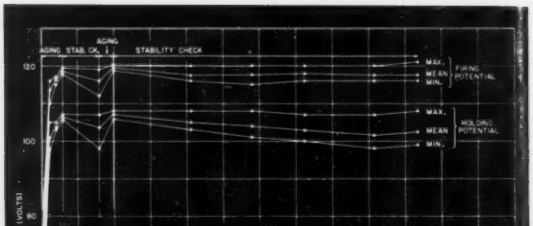
At first only 1 or 2 tubes would fire because they had much lower firing voltages than the others. However, since these drew a heavy current, they soon aged to the point where other tubes took up a portion of the burden. If the characteristic voltage of any tube should drop when processing started, that tube would carry an excess load and quickly raise its firing voltage back to the level of the others. In this way all 10 tubes were soon made to fire simultaneously. Since the

One of the preliminary aging circuits (left) for equalizing and stabilizing gas diode tube characteristics. Later circuits are similar except for using a large number of tubes and for using pulses of different duty cycles. Stability check circuit (right) used at NBS for testing stability of gas diodes during and after the aging process. Tubes are tested under identical conditions, and a separate resistor minimizes mutual interaction between the tubes.









Results of aging tests (left) of type NE–51 cold cathode gas diodes. This is a typical experimental run. Current was kept constant during processing, and processing was continued until the rate of change of characteristic voltage dropped appreciably. A stability check was then begun. Final voltages were found to be reasonably stable as indicated by the curve. Results of another type of aging test (right) on type NE–2 gas diodes, using half-wave 60-cycle current. As each tube aged, its impedance caused the current to drop as test continued. In this case current was allowed to fall rather than be forced to a constant value. A variation in this test was to check for stability before the rate of change of characteristic voltage had dropped considerably. Results of check showed that stability had not been achieved with equalization; however, continuation of the processing followed by another stability check gave results shown in rest of curve, i. e., the final voltages were reasonably stable.

current divided according to the conductances at the holding potential, the characteristic holding potentials tended to become equal because of unequal aging. Thus, equalization of both characteristic potentials was obtained automatically.

Many tubes can be equalized at the same time in this manner, as long as not too much current is drawn by a single or a few tubes. To prevent such an overload, a power supply with poor regulation was used to operate both tubes and relays. A high current load automatically lowered the duty cycle and the pulse voltage. Such protection was especially convenient when the sputtered cathode material formed a conducting path between the electrodes of a tube. In this case the poor regulation of the power supply permitted the path to be evaporated open again without damage to tubes or equipment.

Another method of processing is an automatic "drop-out" technique. Here the source supplying the tubes is a low-voltage pulse whose amplitude is set at the desired final voltage. As the tubes change characteristics, the firing voltage approaches the pulse amplitude, until finally the tube stops firing. In many cases no limiting resistor is needed since the voltage is so low. Variations in current affects the time to drop out, but the final voltage is unchanged.

The use of 60-cycle current as a supply was also investigated. A half-wave rectifier provided the unidirectional pulses. Because of the high duty cycle compared to the pulse method, the current had to be kept to an undesirably low value to prevent overheating, especially at the start of a run, and this increased processing time. Tubes processed in this way behaved much like those processed by relay pulses. The main advantage is the simplicity of the power source.

When a full sine wave was applied to the tubes, however, the results were different. It was found that during the processing the characteristic voltages measured in both directions of conduction changed in a similar manner. However, during a check for stability with unidirectional operation, it was found that the characteristic voltages in the backward direction dropped quickly and became erratic, although those measured in the forward direction seemed to remain fairly stable. This effect is probably due to deposition of material on the anode. When checked for stability with bidirectional operation, the characteristics seemed to be reasonably stable.

Indications are that the improvement over the usual characteristics of the NE–2 or NE–51 is probably due to coating the glass with material sputtered from the cathode. This coating acts as a selective getter, purifying gas in the tube, and as a bulb coating which prevents release of oxygen from the glass. It is to be hoped that sufficient stability for the circuits in which visual indication is required may be obtained with a partial coating. Little sputtering occurs toward the tip of the tube, and no tube has been so blackened that its glowing electrode could not be seen in normal room lighting.

Because of the gas clean-up which occurs, the change in characteristic voltages is tied to the processing. By balancing such things as time and current, it may be possible to control the final voltage to some extent. This depends upon the amount of selective gettering which occurs, the normal gas pressure, and the partial pressure of the undesirable gases inside the tube.

Announcement of New Publications

Computer Development

THIS PUBLICATION presents the more important engineering aspects of the Bureau's electronic computer program. It is based largely on experience relating to SEAC (National Bureau of Standards Eastern Automatic Computer) and DYSEAC (Second SEAC). Topics discussed include dynamic circuitry techniques, systems design, high speed memory development, input-output devices, engineering development, construction and maintenance as well as 3 years' operational experience on SEAC.

SEAC and DYSEAC are high-speed, automatically sequenced, electronic digital computers designed and constructed at NBS. Both machines make extensive use of germanium diodes for all switching and gating. They have mercury delay-line internal memories, and SEAC has an additional electrostatic memory.

DYSEAC represents a considerable advance in the computer art. It was designed to serve as the experimental nucleus for a complex data processing network. Before its delivery to White Sands Signal Corps Agency, DYSEAC was connected directly with SEAC, and the two machines worked cooperatively on a common task for a short time.

At the present time, SEAC continues to be actively employed in the solution of problems at NBS. Its record of productive computation coupled with expanding power and efficiency of operation has proved that it is possible to operate computers for experimental purposes while obtaining useful computations.

National Bureau of Standards Circular 551 is available for $2.00 from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.

Biennial Report

THIS REPORT summarizes the research and development activities of NBS in the physical sciences during the fiscal years 1953 and 1954. Brief descriptions are given of representative accomplishments in each area of the Bureau's responsibilities, which include maintenance of basic standards, determination of physical constants and properties of matter, development of methods and instruments of measurement, and the provision of calibration, testing, and scientific advisory services.

Also included is a discussion of the administrative changes growing out of the recommendations of a special Ad Hoc Committee appointed by the Secretary of Commerce to evaluate the Bureau's program in relation to national needs. For example, a number of technical advisory committees have been established by leading scientific and technical societies to advise the NBS Director in specific technical areas.

During the 2 years, significant results were achieved in programs dealing with electronic computers, electronic instrumentation, and the properties of matter and materials. A new high-speed computer—DYSEAC—which controls and responds to a variety of external devices, was completed and delivered to the Department of Defense. Low-temperature alinement of radioactive nuclei provided new approaches to the study of nuclear disintegration processes. A special ceramic coating was developed for high-temperature protection of alloys in nuclear reactors. Also developed were an electronic instrument that automatically detects changes in a patient under anesthesia, and a remote-control system for automatically measuring radiation intensities and weather conditions in the vicinity of an atomic explosion. Of particular significance was the development of an "X-ray televising" system in which high-energy X-rays may be passed through thick metal objects and then converted to visible light for viewing on closed-circuit television. Also described is a new approach to the design and automatic production of electronic equipment which has excited considerable industrial interest.

A number of advances are also reported in fundamental standards and methods of measurement. To meet the requirement of modern industry, the range of the primary standard of pressure was extended from 50,000 to 200,000 lb/in.^{2} A standard method was set up for calibrating the small, intense sources of beta radiation used in medical treatment. A radiofrequency permeameter was developed to provide an accurate secondary standard of complex permeability for quality control in the manufacture of magnetic materials.

The biennial report is composed of five sections: (1) a general review or summary, (2) a résumé of the Bureau's research and development work in progress or completed during the 1953 and 1954 periods, (3) a review of the testing and calibration program, (4) a discussion of the Bureau's various cooperative activities, and (5) an appendix consisting primarily of statistical and organizational material and a complete list of publications by NBS staff members for the two fiscal years.

National Bureau of Standards Miscellaneous Publication 213 is available for 60 cents from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.

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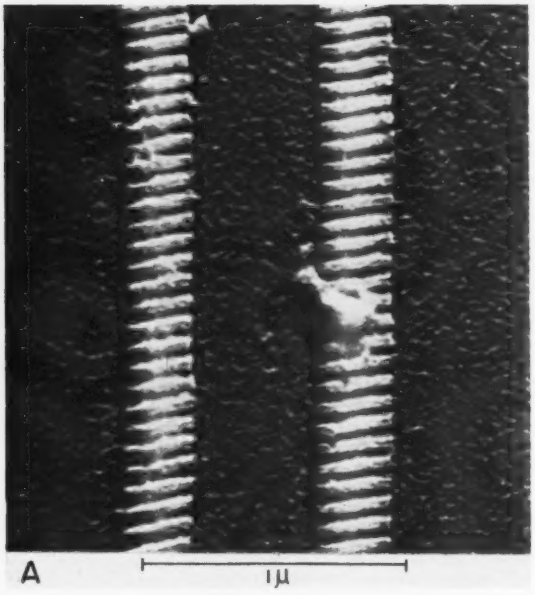

Palladium-shadowed electron micrographs reveal details of collagen structure. A, Characteristic cross-striations in individual fibrils indicate a right-handed helical formation with a helix angle of 9°. B, single swollen fibril. Remnants of the periodic structure seen in A may be observed. In addition, subfibrillar elements make a left-handed helical configuration with a helix angle of about 72°. Geometric discontinuities between subfibrillar elements are assumed to be sites of pores within the fibril.

NEW insight into the structure and behavior of leather should result from pressure porosimeter and electron microscope studies [1] recently completed by R. R. Stromberg and Max Swerdlow of the National Bureau of Standards. The NBS investigation reveals the presence of large numbers of extremely small pores—less than a millionth of an inch in radius—in both leather and its parent substance, collagen. The quantitative information obtained on pore-size distribution within the individual collagen fibrils should aid the leather technologist in understanding the swelling and shrinkage accompanying water-leather interactions. It is also expected to shed additional light on the ability of leather to transmit or absorb water vapor, tannins, or impregnants.

Many of the unique properties of collagen and the tanned product, leather, are believed to result directly from the character of the collagen that is formed in nature as a fibrous network. For example, the remarkable resistance of leather to flexural fatigue may be attributed to the ability of the individual fibers to reorient under stress.[2] Similarly, the permeability of leather to water vapor, which makes it well suited to use in footwear, is related to the presence of pores.

The fibrous structure of collagenous materials is quite complex. Casual examination with the naked eye shows a mass of intertwined fibers. However, when these fibers are examined with the light microscope, they appear as bundles of smaller components known as *primitive fibers*. The primitive fibers, in turn, are found to be made up of *fibrils* when viewed with the electron microscope. Within the fibril, the smallest fiber-like structures thus far resolved by the electron microscope are called *filaments*.

Although considerable overlapping exists, pores in collagenous tissue can be divided roughly into groups corresponding to the structure of the fibrous network. Thus, the largest pores exist between fibers. These probably for the most part have radii larger than 20000 angstroms.[3] The remaining void volume is made up of pores existing between or within the smaller structures. The Bureau's study dealt with pores in the range from 20000 down to 100 A in radius.

The NBS investigation began with a study of pore-size distribution by the pressure porosimeter method.

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[1] *See* Pore-size distribution in collagen and leather by the porosimeter method, *by Robert R. Stromberg, J. Research NBS* **54**, *73 (1955) RP2567; and* Collagen pores determined by electron microscopy, *by Max Swerdlow and Robert R. Stromberg, ibid* **54**, *83 (1955) RP 2568.*

[2] *See* A flex tension test for leather, *by T. J. Carter and J. R. Kanagy, J. Am. Leather Chemists' Assoc.* **49**, *23 (1954).*

[3] *An angstrom is 10^{-4} micron or 10^{-8} centimeter.*

...rein Leather

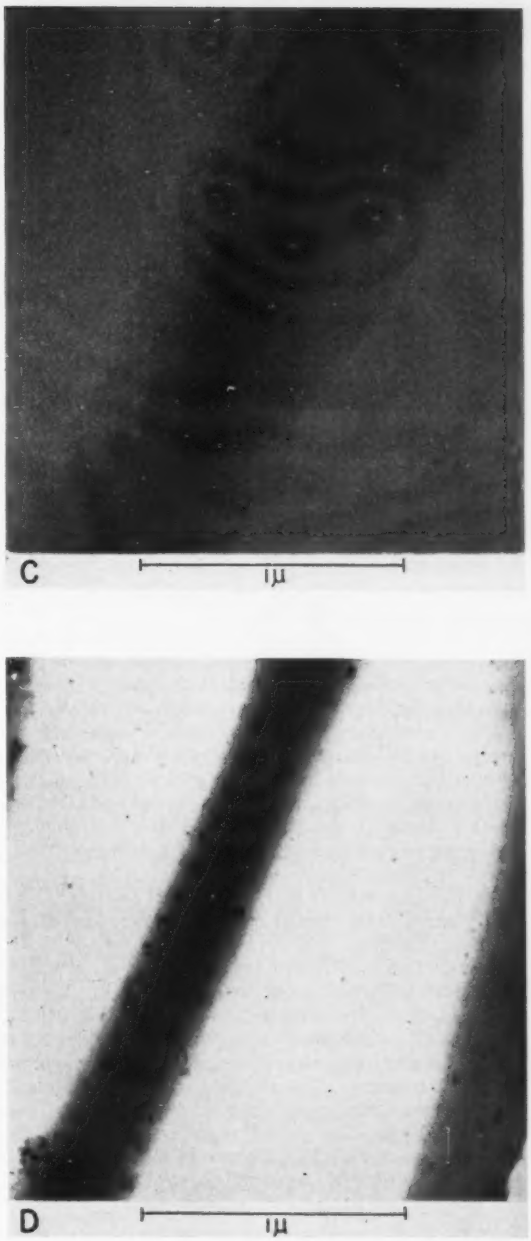
This method is based on the theory, developed by previous investigators, that specified pressures are required to force a nonwetting liquid into pores of certain calculated radii. Thus, a measured quantity of mercury under a known pressure was forced into a previously prepared porous specimen, and the pore-size distribution characteristic of the material was then calculated. Among the materials studied by this method were a group of vegetable-tanned leathers, raw hide, and air-dried kangaroo tail tendon.

The methods of electron microscopy were then employed to confirm the existence of small pores within fibrils and to provide additional information about their size, location, and arrangement in leather and collagen. Kangaroo tail tendon was selected for electron-microscopic study because this material is similar to other mammalian collagen and yields an easily recognized microstructure.

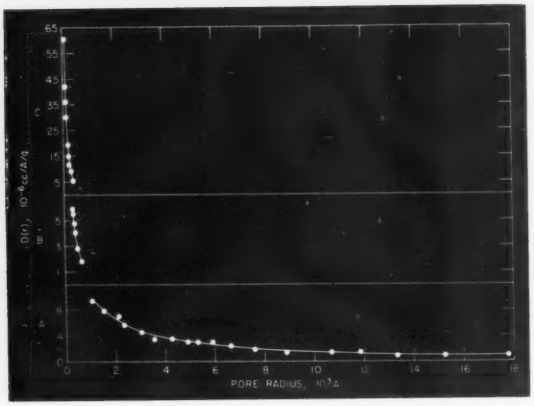
In the pressure porosimeter study, bulk pieces of material were first evacuated to a pressure of about 10^{-5} mm of mercury, then immersed in mercury and subjected to hydrostatic pressures as high as 10,800 lb/in.^{2} for determination of pore-size distributions. In the case of kangaroo tail tendon, this treatment was followed by the preparation procedure required for electron microscopy. Thus, upon removal from the pressure vessel, the tendon was cut into smaller pieces and beaten in clean tap water in an electric food blender for 10 min in order to separate individual fibrils of collagen. Additional specimens of the air-dried tendon which had not been subjected to the mercury pressure treatment were also dispersed for electron microscopy in this way. Drops of the dispersed material were then pipetted onto collodion-covered screens and allowed to dry. To create a three-dimensional aspect and to provide additional contrast, some specimens were shadowed with about 20 A of palladium at an angle such that the shadow length would be about four times the altitude of raised areas of the specimen.

In all the materials investigated by the porosimeter method, two general effects were visible in the plotted data: (1) Mercury was retained within the capillary structure of the porous matrix, and (2) a significant void volume was concentrated in the small pore-size range, 100 to 1000 A in radius. For example, when the volume of retained mercury per gram of specimen was plotted against pressure for the flesh split of degreased vegetable-tanned crust leather, the curve of decreasing pressure did not coincide with the increasing pressure curve. The difference between the starting and end points indicated that the mercury retained by the specimen corresponded to approximately 40 percent of the pore volume explored (ranging from 100 to 18000 A in pore radius).

Once the pore-size distribution had been calculated for a given material, the total amount or percentage of



Electron micrographs of unshadowed collagen fibrils. C, Single, swollen fibril. The open disrupted structure shows the same helical configuration of subfibrillar elements seen in B on opposite page. D, an unshadowed, mercury-impregnated collagen fibril. The dense, elliptically-shaped areas arranged periodically within the fibril are interpreted as mercury-filled pores approximately 150 A in radius. Parallel lines drawn through the dense spots suggest the two helical configurations seen in A, B, and C.



Pore-size distribution curve for air-dried kangaroo tail tendon. Such tendon is considered to be similar to other collagenous tissues used in making leather. Three different ordinate scales are used to make parts of the curve more easily discernible. As with other materials, a significant volume and number of pores were concentrated in the size range from 100 to 1000 A in radius. Note the similarity of the shape of the pore-size distribution curve to that shown in graph on opposite page for finished leather.

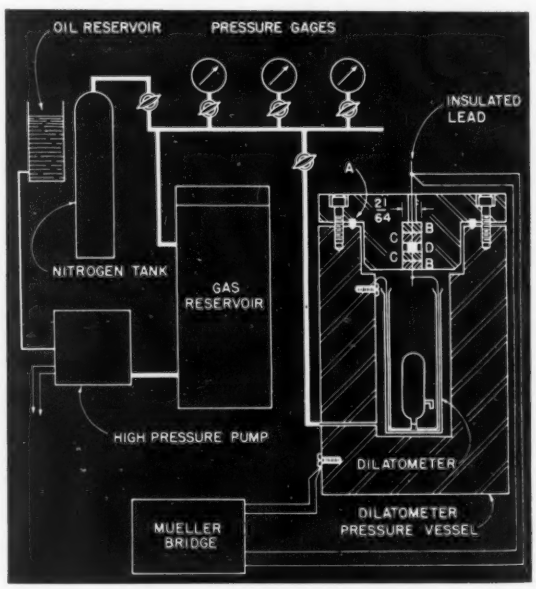
void volume could be determined for selected ranges of pore sizes. Thus, the distribution curve for kangaroo tail tendon showed that of the void volume represented by pores in the range investigated, approximately 75 percent could be assigned to pores ranging from 100 to 1000 A in radius, and approximately 50 percent to pores from 100 to 350 A in radius. This curve showed that for the pore-size range from 1000 to 18000 A in radius, the change in volume with respect to the change in pore radius was small; whereas for the pore-size range from 100 to 1000 A in radius, the pore volume sharply increased as pore radius decreased.

The distribution curve obtained for kangaroo tail tendon showed a close similarity to the curves representing finished sole leather, the corium splits of crust leather, and raw hide. In fact, all the collagenous tissue studied showed a sharp rise in the distribution curve in the region of 100 A in pore radius. It was therefore concluded that a significant void volume must be concentrated in pores of extremely small radii and that this volume must consist of a large number of the small-sized pores.

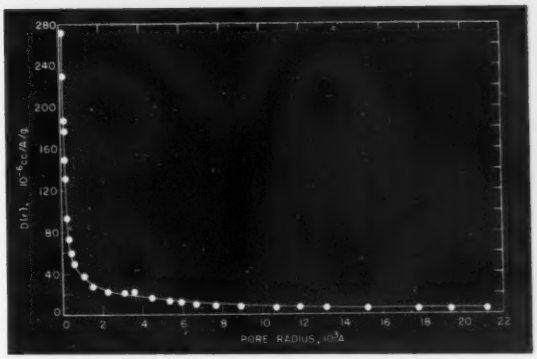
The presence of these small pores was suggested by the electron-microscopical examination of collagen which has not been subjected to mercury pressure but which had been dispersed in the food blender. Many of the fibrils were water-swollen and presented an open, flattened structure. The physical structure of the subfibrillar matrix in these swollen fibrils showed geometric discontinuities which were assumed to be the sites of pores within individual fibrils. However, the expanded subfibrillar matrix was probably distorted, so only an approximation of the shape and size of such pores was possible.

Examination of collagen fibrils obtained from kangaroo tail tendon which had been subjected to mercury under a hydrostatic pressure of 10,800 lb/in.^{2} provided additional information about the arrangement of these small pores within the fibril. The opened, disrupted structure seen in the water-swollen specimens was not observed in any of the specimens which had been impregnated with mercury. However, these fibrils appeared to be differentially "stained" in a periodic pattern of opaque areas. It is believed that mercury became lodged in the pores and retarded the penetration of water into the fibrillar structure. The fibrils impregnated with mercury might then be expected to resist swelling by water and to retain their three-dimensional structure. These findings suggest the use of hydrostatic pressure as an additional variable in the histologic staining and fixing of other types of biologic materials without undue structural distortion.

In the work of other investigators, native collagen from various sources has been generally characterized by a periodic spacing of the order of 640 A along the fibril axis. In the electron micrographs of the undisrupted fibrils obtained from kangaroo tail tendon which had not been subjected to mercury, this characteristic axial periodicity was observed as cross striations with



Mercury pressure porosimeter apparatus used to study pore-size distribution in leather. Leather specimen was placed in the bulb of calibrated glass dilatometer within the pressure vessel. Compressed nitrogen from tank was used to force mercury under a known pressure through the capillary tube of dilatometer and into specimen pores. The quantity of mercury forced into specimen was then determined from the change in electrical resistance of a wire passing through mercury column in the capillary tube. When maximum pressure obtainable from the nitrogen cylinder had been reached (about 2,000 lb/in.^{2}), higher pressures were attained (up to 11,000 lb/in.^{2}) through further compression of the gas by pumping oil into the gas reservoir.



Pore-size distribution curve for vegetable-tanned finished sole leather. Ordinate of the curve is the distribution function in cubic centimeters of pore volume per angstrom of pore radius per gram of specimen. Note that the curve rises sharply in the region of extremely small pore sizes. Area under the curve represents the total pore volume per gram of specimen in range indicated. By the porosimeter method, a pore-size distribution curve is obtained which is characteristic of a given pore population in any material.

spacings varying from 650 to 750 A. These striations seemed to show a helical arrangement and to make an angle of about 9° with the perpendicular to the helical axis, which was also the fibril axis.

In the swollen fibrils the subfibrillar elements also showed a helical configuration, but with a helix angle of about 72°. Remnants of the characteristic periodic spacing could likewise be observed in the disrupted fibrils. It is uncertain whether the helical configurations seen in the electron micrographs were a result of specimen preparation or were a manifestation of the much smaller inherent helical configuration currently proposed for the protein molecule making up the polypeptide chains in collagen.

Corresponding configurations were noted in mercury-impregnated collagen. Thus, when parallel lines were ruled across the fibril axis connecting the opaque areas seen in the micrographs, one set of lines was about 1000 A apart and made a helix angle of about 9°. Another set of lines was about 750 A apart and made a helix angle of about 72°—the angle observed for subfibrillar elements in the swollen fibrils.

The individual opaque areas had an elliptical shape. They were approximately 250 A in one dimension and 350 A in another, the longer dimension being parallel to the fibril axis. In terms of the circular cross section assumed in calculating the pore-size distribution curves, these dimensions would approximate a pore radius of about 150 A. With the apparatus and procedure used in the porosimeter method, the smallest pore size that would be filled with mercury was 100 A. Thus, the electron micrographs appear to provide a visual experimental confirmation of the approximate magnitude and location of pores in collagen fibrils independently deduced from theoretical considerations.

As a result of the Bureau's work, it may be possible to develop more adequate explanations for some of the more significant properties of leather, and thus to make better use of this commercially important material. For example, the transmission of water vapor through leather has been explained on the basis of the porous matrix within the fibrous network. However, present concepts of the mechanism involved will now have to be modified to take into consideration the significant void volume associated with pores of the order of 100 A in radius within the individual fibrils. These pores must necessarily be regarded as additional paths for the transmission of water vapor. When leather is penetrated by water, profound changes take place in its physical properties. It may be possible to explain these changes in terms of the penetration of the water through intrafibrillar pores into zones that are closely associated with the inherent molecular structure of collagen.

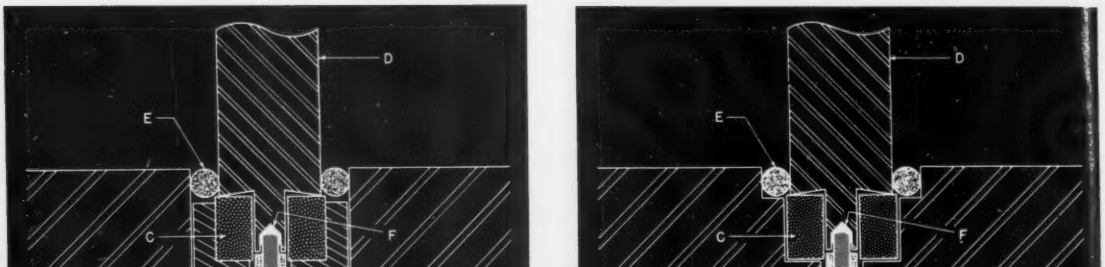
The resistance of leather to abrasion and water penetration has been greatly increased by processes which the Bureau has developed for impregnating leather with rubber and other polymers.[4] These processes depend upon chemical interaction of the leather and the impregnant, but the physical structure of the materials involved must also be considered in the further development of such processes. For example, knowledge of pore-size distribution could be used, in combination with data on the size and shape of polymer particles,[5] to estimate the quantity of material that might enter the porous matrix. With the basic information on the physical structure of leather provided by the Bureau's study the leather technologist should be in a better position to control the extent of impregnation, and thus the properties of the finished product.

[4] *See* Impregnation of leather with synthetic resins, *NBS Tech. News Bull.* **33,** *36 (March 1949)*; Impregnation of leather with natural rubber, *NBS Tech. News Bull.* **33,** *141 (Dec. 1949)*; Butyl-impregnated leather, *NBS Tech. News Bull.* **37,** *44 (March 1953)*.

[5] Electron microscopy of synthetic elastomer latices, *by Robert R. Stromberg, Max Swerdlow, and John Mandel, J. Research NBS* **50,** *299 (1953) RP2419.*

Insulating Seal for High-Pressure Equipment

IN CONNECTION WITH WORK on high-pressure standards, the National Bureau of Standards has devised a special insulating seal which effectively solves the problem of leakage around electrical connections to high-pressure vessels. Simply constructed of inexpensive materials, the high-pressure seal utilizes a sapphire bushing to obtain the necessary combination of high mechanical strength and good electrical insulating properties. The device has successfully withstood pressure up to 170,000 lb./in.^{2} It was designed by H. A.



Combining high mechanical strength with good electrical insulating properties, the NBS high-pressure insulating seal (left) withstands pressures up to 170,000 lb/in.² A, Closure plug body; B, soft steel pad; C, insulating bushing; D, soft steel electrode; E, rubber O-ring; F, soft solder joint; G, copper wire from pressure measuring coil; H, spaghetti insulation. *Right:* A simplified version of the high-pressure seal in which use of a softer bushing permits elimination of the soft steel pad.

Bowman and associates of the Bureau staff working under the sponsorship of the Army Ordnance Corps.

Methods of measuring and controlling high pressures are becoming increasingly important to science and industry. For example, in the chemical industry, pilot-plant operations are now being run at pressures as high as 100,000 lb/in.², and laboratory operations are moving into the range from 100,000 to 200,000 lb/in.². Also, in ordnance, both laboratory studies and large-scale operations are making use of very high pressures. As pressures increase, problems of equipment design become increasingly difficult. Not the least of these has been the problem of making electrical connections to the interior of a high-pressure vessel without leakage.[1] Though developed specifically for use in calibrating high-pressure standards, the Bureau's high-pressure seal may prove useful in chemical, metallurgical, petroleum refining, and other industrial equipment where high-pressure operations are monitored or controlled electrically.

Above 50,000 lb/in.² hydrostatic pressures are commonly measured by inserting a previously calibrated resistance coil in the pressure vessel and noting the change in resistance as pressure is applied. In commercial equipment, one end of the pressure-sensitive resistance coil is usually attached to a high-pressure steel closure plug inserted in a hole in the pressure vessel. The other end of the coil is attached to an electrode which is inserted in the central opening of the closure plug but which is insulated from the plug by a bushing of some kind. External connections can then be made to the insulated electrode and the exposed portion of the steel closure plug. To minimize the problem of changes in lead resistance, a 4-lead system is usually employed for precision measurements.

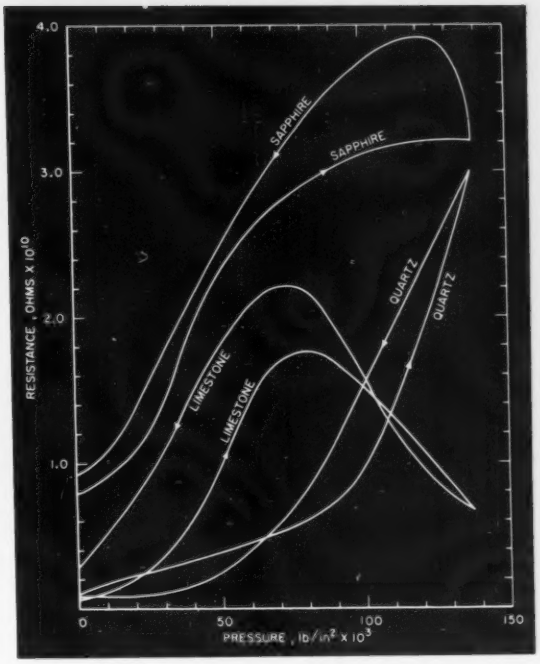
In order to utilize commercial equipment as far as possible, the Bureau's insulating seal was designed so that the space occupied by a 4-lead connection would be the same as that required for commercially available single-wire fitting of the same shape. It was also decided that the resistance of the insulating bushing should be not less than 1,000 megohms within the pressure range under consideration.

The NBS seal is composed of a soft steel electrode, a sapphire or quartz insulating bushing, a rubber O-ring, and a soft steel pad, assembled and set into a cavity in the closure plug. The soft steel pad fits directly into the plug cavity and supports the insulating bushing. Through the hole in the bushing passes the electrode, to which is soldered one end of the pressure measuring coil. The portion of the electrode external to the bushing is larger in diameter than the internal part and presents a tapered surface to the external face of the bushing. As a result of the taper, only the outer rim of this surface makes contact with the bushing unless very high pressure is applied. The rubber O-ring separates the larger-diameter portion of the electrode from the wall of the cavity in the closure plug.

In operation the seal acts in two stages. The O-ring provides the initial low-pressure seal through its compression contact with the electrode and the interior wall of the cavity in which the assembly is installed. This seal probably lasts up to about 30,000 lb/in². Then, at about the pressure that the O-ring becomes ineffective, the tapered section of the electrode is deformed against the bushing, thereby sealing off the leakage path between the bushing and the electrode. Simultaneously, or shortly thereafter, the soft steel pad is deformed by pressure, sealing off the leakage path between the bushing and the wall of the plug cavity.

Commercially available sapphire instrument bear-

[1] The physics of high pressure, *by P. W. Bridgman, p. 51 (G. Bell & Sons, Ltd., London, 1949).*



Curves showing insulation resistance of sapphire, quartz, and limestone bushings as functions of applied pressure. Shape of these curves is thought to be due, at least in part, to distortion of the rubber O-ring as high pressures are applied. Upper limits of the curves were dictated by safety factors in other parts of the apparatus, rather than by the strength of the bushings themselves.

ings have proved quite satisfactory as insulating bushings. Besides having the required physical characteristics, they are inexpensive and can be obtained already ground to extremely exacting tolerances. It has been found that when the basal plane of the crystal becomes tangent to the planes of shear force in the loaded bushing, the crystal slips along this plane, probably because of twinning. However, this slippage does not interfere with the operation of the seal since the steel pad is again deformed against the interior surface of the bushing after slippage. Selection of bushings with the proper crystal orientation is expected to reduce the possibility of slippage. Fused quartz is less brittle than sapphire and is almost as good an insulator.

The high-pressure seal can be simplified considerably by choosing softer bushings that do not require the soft steel pad. With this type of seal, bushings of lithographic limestone have been used successfully. However, great care must be given to the selection of the limestone since this stone has many flaws which may cause the bushings to crumble under load. Once a flawless piece has been selected, it is easily turned on the lathe using sharp tools, preferably carbide, and taking small cuts.

Selection of Bolt Materials for Underground Service

A RECENT STUDY by the Bureau has shown that electrical measurements made in the laboratory can be generally useful in predicting the corrosion resistance of bolt materials used underground.[1] The study, conducted by W. J. Schwerdtfeger of the NBS corrosion laboratory, is an application of earlier work which demonstrated that general corrosion rates can be measured electrically.[2] The present laboratory study shows that some bolt materials can be protected against corrosion by making them cathodic to the structures which they fasten.

Although failures of plain ferrous bolts underground are confined chiefly to severely corrosive environments, the costs attributed to shutdowns and repairs are high. When iron or steel is exposed to the soil, local differences in electrical potential develop at the surface of the metal, resulting in the formation of numerous small corrosion cells. As a consequence, electric currents flow through the soil from certain areas (anodes) to areas of less negative potential (cathodes), with accompanying loss of metal from the anodes. If a bolt becomes anodic to a structure, it will fail more rapidly than the surrounding metal because of the comparatively large differences in area.

Presumably, bolts and structures made of the same materials should corrode at the same rate. However, it is believed that the many sharp edges on the surface of bolts have an accelerating effect on the corrosion rate. In addition, when the soil around the bolt is packed more tightly than the earth around the adjacent structure, corrosion may be further accelerated because of increased galvanic action.

Other workers[3] have found that the addition of 1 percent copper to cast-iron bolts resulted in a marked reduction of corrosion on the bolts when coupled with plain cast iron. The work of these investigators also suggested the use of a small amount of nickel in ferrous bolts. As extensive use of low-alloy bolts would

[1] Electrical measurements in the selection of bolt materials for service underground, *by W. J. Schwerdtfeger, J. Research NBS* **52**, 5 *(1954)*.

[2] Measurement of the corrosion rate of a metal from its polarizing characteristics, *by W. J. Schwerdtfeger and O. N. McDorman, J. Electrochem. Soc.* **99**, *407 (1952)*; *and* Electrical measurement of corrosion rate, *NBS Tech. News Bull.* **37**, *22 (Feb. 1953)*.

[3] Control of galvanic corrosion of iron, *by C. K. Donohoo and J. T. MacKenzie, Corrosion* **2**, *20 (1946)*.

Gland sections were cut from 4-in. cast-iron pipe.

not result in prohibitive installation costs to industry, such bolts were arbitrarily chosen for the main laboratory study. High-alloy cast-iron and nickel-copper alloy (70 to 30) bolts, known to be cathodic to plain cast iron but relatively expensive, were also used.

In order to determine what electrical measurements might be of value in predicting the probable behavior of bolts for use underground, potential measurements were made on the components of galvanic couples (bolts joined with sections of pipe), followed by exposure of the couples to a very corrosive soil for 1 year in the laboratory. Potentials and currents were measured during the exposure period, and weight losses were obtained at the conclusion of the period.

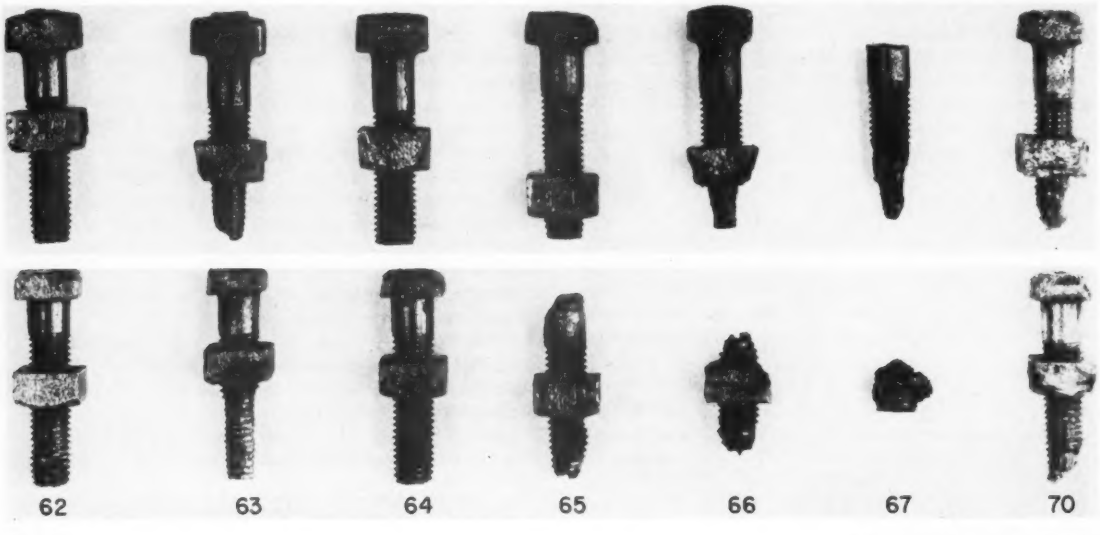
The principal measurements were made using sections of pipe glands bolted together and buried in a highly corrosive soil. The sections were cut from 4-inch cast-iron pipe glands (components of mechanical joints). The bolts were insulated from the glands and then electrically coupled by tapping copper wires into the glands and bolts. The copper wires could be separated for making electrical measurements but were directly connected during the rest of the study. Galvanic currents between the components of the couples and the open-circuit potentials of the components were measured biweekly during the initial 4 months of exposure, and monthly thereafter. Before disassembling the couples for cleaning and weight-loss measurements, cathodic polarization curves were obtained on each bolt.

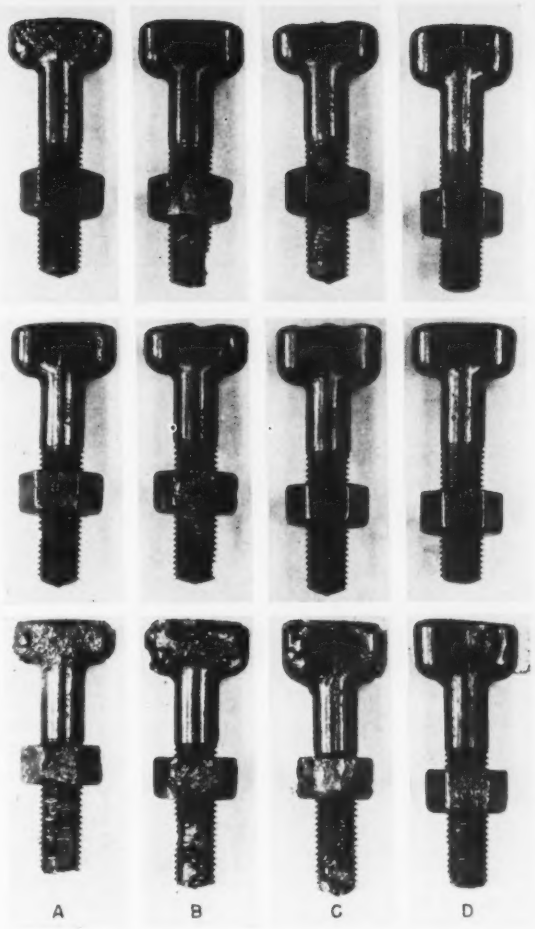
The reduction of the corrosion on the bolts resulting from their being cathodic to the gland sections was found after the weight losses were measured (cols. 3 and 4, table 1). The reduction in weight loss of the coupled bolts based on the uncoupled controls ranged from 53 to 93 percent. The data indicated the beneficial effects of relatively small amounts of alloying constituents, particularly 1.5 to 2 percent of nickel. The cathodic nature of high-alloy cast-iron and nickel-copper alloy resulted in almost negligible corrosion of these bolts. Complete cessation of corrosion of the low-alloy bolts was not expected because the gland sections furnishing the current limited the potentials assumed by the bolts. This prevented a drift in potential of the bolts to the protective value.

The electrical measurements were correlated with weight losses at the end of the investigation. It was found that, for bolts made of materials having similar rates of corrosion, larger galvanic currents generally provided for better protection of bolts. This was shown by larger open circuit potentials measured during exposture and correspondingly lower weight losses.

The effect of exposure time on the corrosion rates of the various bolts was also studied by using the cathodic polarization curves taken before removal of the bolts. In general, at the conclusion of the laboratory exposure all of the bolts were corroding at a lesser rate than previously (col. 5, table 1).

Bolts removed from couples after an underground period of 13 years at the 14 NBS test sites. Each pair of bolts, cast iron (upper row) and steel (lower row), is shown above the number of the site at which it was buried. Each pair of bolts (0.625 by 3 in.) joined two cast-iron plates (0.5 by 3.5 by 12 in.) that overlapped by 1.5 in.





Cast-iron bolts exposed to very corrosive soil for 1 year in the laboratory at NBS. A, cast iron (1 Cu); B, cast iron (1 Ni, 1 Cu); C, cast iron (2 Ni, 1 Cu); D, high alloy cast iron. Bolts in the upper two rows were coupled to gland sections. Bolts in lower row are uncoupled controls. Parts of the controls not normally in direct contact with the soil were coated with bituminous paint.

An effect that also had to be taken into account was the possible corrosive action of the cathodic bolts on the anodic gland sections. To compare the effect of normal corrosion on a gland section with the effect of the corrosion attributed to the cathodic bolts, one couple was left on open circuit throughout the exposure period. Even though galvanic current between a cathodic bolt and a structure will produce weight loss in accordance with Faraday's law, the laboratory data show that local action currents on a portion of the structure are reduced by the galvanic current. Because of the relatively larger structural area of actual service installations, the over-all effect probably would not be detrimental.

An additional study was also made of the probable effect of replacing bolts in cast-iron structures in which graphitization has occurred. In this work two specimens were cut from pipe sections that had been exposed to corrosive soil for 13 years and had become graphitized. These were exposed in the laboratory for about 9 months along with two previously unexposed cast-iron specimens. Upon initial exposure the graphitized specimens were cathodic to the ungraphitized specimens by as much as 400 millivolts, but for the final 60 days of exposure the potential difference averaged about 15 mv. Even though this potential difference appears to be small, plain ferrous bolts used as replacements on cast iron structures would corrode rather rapidly because of the area relationship. However, steel containing 5 percent of chromium was found to be cathodic to unalloyed ferrous material by about 50 mv, indicating that it might be a desirable bolt material, especially for replacement purposes.

TABLE 1. *Comparison of weight losses of uncoupled and coupled bolts*

Bolt material	Weight loss of bolt [a] after 368 days of exposure		Reduction in weight loss of coupled bolts based on control (uncoupled)	
	Control uncoupled	Coupled cathodic	Based on the actual weight loss	Based on the [b] calculated corrosion rate at the end of the exposure period
	g	*g*	*Percent*	*Percent*
Cast iron (1 Cu)	28.5	5.3	81.4	89.5
Cast iron (1 Ni, 1 Cu)	25.6	9.1	64.4	88.7
Cast iron (2 Ni, 1 Cu)	33.8	3.5	89.7	97.9
Plain carbon steel	16.4	7.6	53.7	56.7
Cr-Ni-Cu-Si steel	19.7	7.1	64.0	77.7
Ni-Cu steel	14.2	3.8	73.2	71.9
High-alloy cast iron	8.8	0.61	93.1	>95
Nickel-copper alloy	0.95	.19	80.0	>95

[a] Average to 2 bolts.
[b] Weight loss (grams) = KtI, where $K=2.8938\times10^{-4}$ g/coulomb; $t=368$ days, expressed in seconds; and $I=0.83\ I_p$ in amperes.

Publications of the National Bureau of Standards

Journal of Research of the National Bureau of Standards, volume **54,** number 4, April 1955 (RP2579 to RP2585 incl.). Annual subscription $4.00.

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A. V. ASTIN, *Director*

May 1955 **Issued Monthly** **Vol. 39, No. 5**

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Some applications of extreme-value methods. E. J. Gumbel and J. Lieblein. Amer. Statistician (1108 16th St. NW., Washington, D. C.) **8**, No. 4, 14 (Dec. 1954).

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